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DETECTING AND MEASURING MECHANICAL PICKER LUBRICANTS ON COTTON[1/]

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Since the mechanical cotton picker became widely accepted by cotton producers, research workers and textile manufacturers have been concerned about the possible harmful effects of lubricating oil contamination of the cotton by the machine. In 1958 it was theorized that spindle lubricating oils had contributed to the failure of one replication (32 bales) of a particular field test at Stoneville, Miss., to spin properly in the mill. The test was repeated in 1959. Picker servicing was carefully supervised and ultra-violet light was used in the field to visually inspect each bale for excessive oil contamination. Any bale that appeared to have an unusual amount of oil was removed from the test. As a result, no difficulty was encountered in spinning.

Because the 1959 test did not prove that oil contributed to the failure of the 1958 test, additional laboratory and field experiments dealing with oil contamination and its effects on cotton spinning were planned. Since the study required the measurement of minute quantities of oil, the need for a quick and simple method of measuring these small quantities in the field was soon apparent. This paper describes the development of rapid methods of measuring oil on cotton in the field.

Mechanical cotton pickers using tapered spindles employ two systems of spindle bar lubrication: (1) Grease-packed bars lubricated at 20- to 30-hour intervals, and (2) bars flushed each night with a nondetergent, light-grade oil. Work reported here deals only with oil. Grease contamination investigations are underway.

1/ Cooperative investigations at the Delta Branch, Mississippi Agricultural Experiment Station, Stoneville, Miss., which are part of a contributing project to Regional Cotton Mechanization Project S-2.

2/ Agricultural Engineers and Cotton Technologists, respectively, Agricultural Engineering Research Division.

TECHNIQUES FOR MEASURING OIL ON LINT COTTON

Two principal methods are used to measure oil on lint cotton--quantitative and qualitative. The quantitative method, until now, has consisted of only one technique--reflux extraction of the oil. With the information obtained from current research, the detection of oil by fluorescence can also be done quantitatively. Visual observation of oil on lint cotton under an ultraviolet light provides a qualitative analysis of the degree and distribution of oil contamination. Each of these techniques is fully explained.

Reflux Extraction

The reflux extraction principle for measuring oil on lint cotton has been used in previous tests dealing with oil contamination. Duplicate 5-gram samples are wrapped in #7760 filter paper and placed in a filter tube, which is inserted in a flask of known weight. By a continuous process of evaporation and condensation, the solvent (Skellysolve-B, a petroleum ether) is allowed to pass through the sample for 2 hours. The remaining solvent is then evaporated, leaving only the oil residue in the flask. The gain in the weight of the flask is reported as the amount of petroleum oil, natural oils, waxes, and gums extracted from the lint cotton. The natural oils, waxes, and gums so extractable are compensated for by control lots.

By actually weighing the oils that were on the cotton, any change in lint moisture from the initial moisture content does not affect the final calculation. Also, based on actual checks, the filter paper used to hold the cotton together does not affect the final oil analysis.

The petroleum ether employed (Skellysolve-B) evaporates at a much lower temperature than the oils on cotton do. Even though some residue is left from the ether, it is so minute that it does not affect the final analysis. Approximate cost for analysis per sample is $2. Results are reported in percentage of oil by weight of lint cotton.

Quantitative Analysis Through Fluorescence

The fluorescence phenomenon is serving mankind in many fields. Agriculture, biology, crime detection, food inspection, and medical professions are using this tool. Excitation of the fluorescent material is provided by lamps emitting energy in the ultraviolet region. Both qualitative and quantitative analysis techniques employing flourescence have long been used to solve problems in some branches of science. Radley and Grant (6)[3/] have outlined the uses of fluorescent analysis and included extensive bibliographical material on the subject.

3/ Underscored numbers in parantheses refer to literature cited, page 9.

The petroleum industry has long used the fluorescent properties of oil to expedite their drilling operations. Very dilute solutions of mineral oils fluoresce under ultraviolet lamps ranging from 2,000 to 3,600 angstroms. DeMent (3) states that the fluorescent colors of samples vary unless they come from identical samples. Colors normally range from light to dark blue. Sussky[4/] reports that it is believed that oil fluorescence is caused by the presence of aromatic and unsaturated compounds, since saturated hydrocarbons do not fluoresce.

Fluorometric analysis. Measurements are necessary to obtain the ranges of oil contamination that might affect spinning. Two methods of quantitative analysis were investigated. These were fluorescence measurements with a fluorometer and reflux extraction with petroleum ether, which was described previously.

Recently developed fluorometers measure the radiant energy emission of fluorescent substances that are excited by ultraviolet lamps. Early fluorometers were equipped with a logarithmic scale; however, recent investigations by Yoe and Koch (7) have shown that the fluorescent intensity of a dilute solution is a straight-line function of concentration. Liljedahl and Straight (5) used a similar instrument to measure spray deposit concentrations tagged with fluorescent materials.

Fluorometer. The fluorometer used was a Turner Model 110.[5/] This instrument is essentially an optical bridge which measures the difference between light emitted from the sample and that from a calibrated rear light path. A balanced light condition between the sample and rear light path is indicated by the null position of the meter. Light required to balance the bridge is indicated on the graduated fluorescence dial. Interchangeable filters are used to allow light transmission at the wavelengths required for use with the fluorescent material.

Data reduction with this instrument is simple and rapid. Known samples are eluted in the appropriate solvent, a portion of the liquid is poured in a cuvette, and the readings are recorded on a calibration curve.

Phillips[4/] used redistilled Skellysolve-B for a solvent to extract oil from cotton. The oil contamination of the cotton was in the order of 145 parts per million. A large error was observed, attributable to background fluorescence of the solvent. His suggestion was to try either heptane or hexane for higher concentrations of oil.

4/ Private communication.

5/ Appreciation is expressed to Dr. R. E. Phillips of Turner Associates for selecting solvents and filters for tests.

The range of oil concentrations used for knowns was from 1,800 to 9,000 p.p.m. Five-gram samples of cotton were chosen as a convenient size. Two hundred ml. of heptane was used to elute each sample. Samples of heptane were run in the instrument to establish the background fluorescence level of the solvent. Then the series of cottons were run with known concentrations to establish a calibration curve for the instrument. The laboratory setup is shown in figure 1. The rugged construction of the fluorometer permits oil contamination determinations to be made in the field, if required.

Figure 1. Laboratory setup for oil determination on lint with fluorometer. Five-gram samples of cotton are weighed on torsion balance. Samples are then eluted in 200 ml. of solvent. Solvent is poured in cuvette, which is inserted in fluorometer, and meter reading is recorded. Values are then assigned from calibration curve made from known values.

Qualitative Analysis Through Fluorescence

Use of ultraviolet light to detect mineral oil stains on textile products is an established practice. A light-tight cabinet was constructed for observing oil on machine-picked cotton (Fig. 2). The cabinet contained two 15-watt black light tubes powered by a converter that plugs into the cigarette lighter socket of a pickup truck. Figure 3 shows this cabinet placed against the open door of a cotton picker. A 35-mm. camera is shown in the view port. Photography of fluorescence is easily accomplished in either black-and-white or color. Covering the camera lens with simple filters prevents fogging of the film (1, 2).

This cabinet has been used for observing the inside of the picker drum after various types of service. Quick visual checks were made on the amounts of lubricants deposited on seed cotton after service.

Figure 2. Portable black light cabinet for viewing oil deposits on machine-picked cotton. Cabinet does not have bottom and is moved across cotton in the trailer.

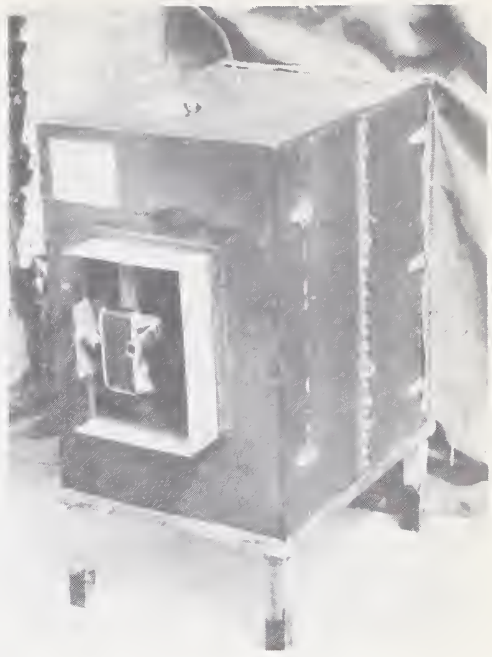


Figure 3. Portable black light cabinet set up to view inside of picker drum. Light cracks are covered with tarpaulin. A 35-mm. camera is mounted in view port for photographing oil distribution in drum.

RESULTS

Accuracy of the Reflux Extraction Method

Checking the accuracy for measuring oil on lint cotton involved treating samples of hand-picked lint cotton with varying known weights of oil. The oil, which weighed 0.87 gram per milliliter, was applied to the lint cotton by a pipette calibrated to deliver 0.01-ml. increments. The samples, all of which weighed 5 grams after the oil was added, were then processed by the reflux extraction method.[6/]

The average percentage of error of the reflux extraction method for various concentrations of oil on lint is given in table 1. The error ranged from 0.6 percent for 0.174-percent oil concentrations to 20.7 percent for 0.348-percent concentrations. Figure 4 shows how the four replications deviated from the known concentration curve. The absolute accuracy of the reflux extraction method cannot be calculated from the information in this report because the actual variation in amount of vegetable oil among untreated samples of lint cotton is unknown. For example: from 0.30 percent to 0.42 percent of vegetable oil was extracted from samples of hand-picked, untreated lint cotton. This variation could have been caused either by an error in the extraction method or by variations in actual percentage of vegetable oils on the samples.

6/ Appreciation is expressed to D. A. Bradham, Analytical Chemist, Barrow-Agee Laboratories, Inc., Greenville, Miss., for working up the reflux extraction method and for his cooperation in running the tests described in this report.

On the other hand, up to ± 0.066 percent of lubrication oil added to lint cotton can be accounted for by using the reflux extraction method of determining oil contamination of lint cotton. The fact that the variation in amount of vegetable oil on the untreated samples was 0.12 percent and the standard deviation from regression (fig. 4) was ± 0.066 percent, or a total of 0.132 percent, indicates that the average error of the extraction method itself may have been only 0.012 percent of oil. Thus, the reflux extraction method is very accurate in determining the percentage of lubricating oil on lint cotton. In general, the error in the method itself is often less than the variability in the amount of natural oils on the untreated lint samples.

Table 1. Accuracy of the reflux extraction method for measuring oil on lint cotton, Stoneville, Miss., 1962[1/]

Oil applied on 5 grams of lint cotton[2/]	Oil extracted[3/]	Calculated oil added	Error
Percent	Percent	Percent	Percent
0	0.350	0	--
0.174	.525	0.175	+0.6
.348	.770	.420	+20.7
.519	.880	.530	+2.1
.691	1.070	.720	+4.2
.863	1.235	.885	+2.5

1/ Extracted by Barrow-Agee Laboratories, Inc., Greenville, Miss.
2/ SAE 10 nondetergent oil.
3/ Average from 4 samples.

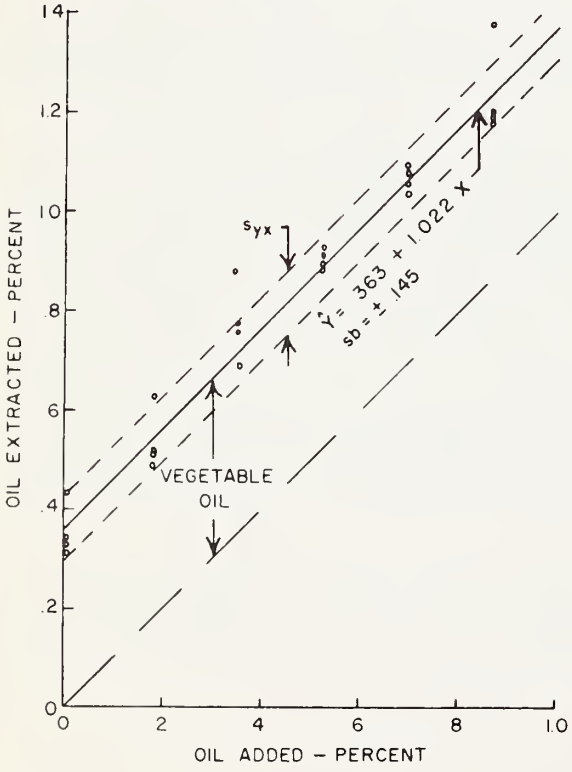


Figure 4. Accuracy of the reflux extraction method for measuring lubricating oil on lint cotton.

Accuracy of the Fluorometric Analysis Method

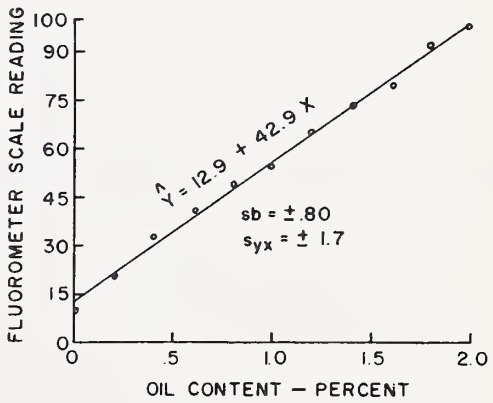
Determining a correction factor. Since SAE 10 nondetergent oil is recommended for lubricating the spindle bars of the mechanical picker, this grade oil was used for all the tests.

Oil was added to 200 ml. of heptane in 0.01-gram increments up to 0.1 gram. The solution was then agitated and a sample of it was poured into a cuvette which was inserted in the door of the fluorometer. Fluorescence readings were then recorded for the various samples of known oil concentrations.

The oil concentrations were converted to percentage of oil on 5 grams of lint cotton, assuming that the 5-gram sample of lint cotton was eluted in the 200 ml. of heptane. More precisely, if 5 grams of lint cotton containing 1 percent of oil (0.05 grams) were eluted in 200 ml. of heptane, the heptane would then contain 0.05 gram of oil. The same solution of oil in heptane could be made by adding the 0.05 gram of oil directly to the 200 ml. of heptane.

The fluorometer readings vs. known oil concentrates are presented in figure 5 as a straight line relationship. Moreover, the equation of the straight line is $Y = 12.9 + 42.9X$ where Y = fluorometer 30X scale-reading on oil-heptane concentrate, X = percentage of oil that would be extracted from 5 grams of lint cotton, and the constant (12.9) is the fluorometer reading on the heptane alone. The standard error of the regression coefficient was found to be $\pm$ 0.80 and the standard deviation from regression $\pm$ 1.7. The fluorometer reading on heptane alone varied somewhat when the source of supply was changed. This indicates that a reading on heptane alone must be made when the supply tanks are changed.

Figure 5. Fluorometer reading vs. percentage of oil contamination of lint. Curve holds true only when 5 gm. of lint is eluted in 200 ml. of heptane, and light intensity is read using 2A plus 2ND secondary filters and 7-60 primary filter in the fluorometer.



Accuracy of the correction factor. As a check on the accuracy of measuring the oil on lint cotton by fluorometer analysis, hand-picked samples of lint were treated with known weights of oil. These samples, along with an untreated check, were then eluted in 200 ml. of heptane and meter readings were recorded for the different heptane-oil concentrations. The average meter reading for the check was subtracted from the average meter readings for the treated samples and the remainder was multiplied by the previously determined correction factor (1/42.9 = 0.0233) to get the computed percentage of oil on the lint. As listed in table 2, the error in computing the percentage of oil on lint cotton by fluorometric analysis ranged from 0.4 percent to 12.1 percent.

Table 2. Accuracy of measuring oil on lint cotton by fluorometric analysis

Actual oil added	Total light reading[1/]	Light reading on SAE 10 oil[2/]	Computed oil added[3/]	Error
Percent			Percent	Percent
0	8.25	0	0	--
0.174	16.62	8.37	0.195	+12.1
.348	24.00	15.75	.367	+5.5
.519	30.62	22.37	.521	+.4
.691	40.50	32.25	.752	+8.8
.863	47.75	39.50	.921	+6.7

1/ Average scale reading from 4 samples.

2/ Light intensity caused by the added SAE 10 oil was obtained by subtracting from the total light intensity (column 2) the light intensity caused by the untreated cotton (8.25).

3/ Computed by dividing the light reading on the SAE 10 oil by the regression coefficient 42.9.

In this test, the fluorometer reading on heptane alone ranged from 5.5 to 6.0 and the reading on heptane that had been used to elute 5 grams of untreated lint cotton ranged from 8.0 to 10.0. Therefore, the heptane dissolves some of the vegetable oil from the lint. It is also recognized that a portion of the error in the fluorometric analysis method is caused by fluctuations in the amount of vegetable oil that is dissolved in the heptane between lint samples.

The fact that all error computations in table 2 were positive indicates that a higher constant should have been subtracted from each fluorometer reading. Moreover, when a regression curve showing fluorometer reading versus percentage of oil added was calculated, the point of intersection of the curve with the Y-axis was found to be 8.41, which was slightly higher than the average reading (8.25) on the check samples. Therefore, most of the error in computing oil content by the fluorometric analysis method is caused by variability in amount of oil on lint samples and not by instrument error.

CONCLUSIONS

The evaluation of cotton contamination with oils depends on rapid methods of measuring oil on the cotton. This is essential in controlled test work.

A black light view box is a valuable aid for field observations of picker cleanliness. Seed cotton can be easily examined in the trailer.

Two methods of quantitative analysis can be used--reflux extraction and fluorometric analysis. The method of fluorometric analysis was accurate, rapid, and simple. Fluorometric analysis is accurate with ± 10 percent, and the error can be further reduced by careful handling.

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